

National Aeronautics and Space Administration

NASA CR-174915 R85AEB307

NASA-CR-174915 19850018400

# EFFECTS OF SURFACE CHEMISTRY ON HOT CORROSION LIFE

## **SECOND ANNUAL REPORT**

By

R.E. Fryxell

General Electic Company
Aircraft Engine Business Group
Cincinnati, Ohio 45215

LIBRARY COPY

June 1985

1505

LANGLEY RESEARCH CENTER LIBRARY, NASA HAMPTON, VIRGINIA

Prepared for

**National Aeronautics and Space Administration** 

NEO 1 2 1 7

Lewis Research Center Contract NAS3-23926

1. Report No. NASA CR-174915	2. Government Acces	sion No.	3. Recipient's Catalo	g No.				
<u> </u>	4. Title and Subtitle							
	5. Report Date June 19	85						
Effects of Surface Chemistry on	6. Performing Organization Code							
7. Author(s)			8. Performing Organia	ration Report No.				
D E E								
R. E. Fryxell			10. Work Unit No.					
9. Performing Organization Name and Address								
General Electric Company		· · · · · · · · · · · · · · · · · · ·	11. Contract or Grant	No.				
Evendale, Ohio 45215-6301			NAS3-23926					
			13. Type of Report a					
12. Sponsoring Agency Name and Address			Second Annua Contractor					
National Aeronautics and Space Washington, D.C. 20546	Administration		14. Sponsoring Agency					
15. Supplementary Notes								
Project Manager, Nathan S. Jac Cleveland, Ohio	obson, NASA Lewi	s Research Center,						
16. Abstract								
cycling, 0.5 ppm sodium as Nat 700, René 80, uncoated and wi 1000 hours, have been complete at 1100°C in oxidizing or in- changes used for periodic non in following the course of con Typical sulfidation was observed service-run turbine components life of RT21 and Codep coating NiCoCrAlY coating. The exten- on U700 substrates than on Re- result of composition changes surface oxidation.	th RT21, Codep, of the RT21, Codep, of the section of the RT21, Codep, of the RT21, Co	or NiCoCrAlY coating in the as-processed for times up to 600 pection of specimens of structurally similes of caused a severe cant but less decreases was much greate coating life decr	s. These tests, condition and a hours. Coil in have been found ar to that obser decrease in hot se in the life or for all three ease is predomin	up to fter aging ductance useful  ved for corrosion f the coatings antly a				
17. Key Words (Suggested by Author(s))	achter i felicitimuseus as anner anticititimuseus as annes annes a <u>n annes anticitimus</u> es com	18. Distribution Statement						
Hot Corrosion		Unclassified -	Unlimited					
Gas Turbine								
Life Prediction Methodology		-						
	•							
19. Security Classif. (of this report)	20. Security Classif. (c	of this page)	21. No. of Pages	22. Price*				
Unclassified	,	, -	41					
Auctassitien	Unclassified		<u> </u>					

<sup>\*</sup> For sale by the National Technical Information Service, Springfield, Virginia 22161

#### FOREWORD

This Second Annual Report, covering the period 2 May 1984 to 2 May 1985, was prepared by the Engineering Materials Technology Laboratories (EMTL) of General Electric's Aircraft Engine Business Group, Cincinnati, Ohio, 45215, under NASA Contract NAS3-23926. R.E. Fryxell of General Electric is the principal investigator and N.S. Jacobson of NASA-Lewis Research Center is the Project Manager for NASA.

This program involves extensive collaboration, under subcontract, of B.K. Gupta, C.S. Kortovich and Gail E. Leese at the Materials and Manufacturing Technology Center of TRW Inc., Cleveland, Ohio, 44117.



# TABLE OF CONTENTS

	Page
SUMMARY	1
INTRODUCTION	3
TASK II - BASELINE HOT CORROSION TESTS	5
Results	7
TASK III - AGING TREATMENTS	16
Results	16
TASK IV - HOT CORROSION TESTS OF AGED SPECIMENS	27
CONCLUSIONS	40
REFERENCES	41

## LIST OF ILLUSTRATIONS

Figure		Page
1.	Changes in Coil Inductance with Hot Corrosion at 900° C, Cyclic Exposure.	10
2.	Photomicrographs from Burner Rig Hot Corrosion Tests at 900° C.	11
3.	Photomicrographs of NiCoCrAlY Coated Specimens from Hot Corrosion Burner Rig Test at 900° C Showing Progressive Degradation with Time.	14
4.	Photomicrograph of NiCoCrAlY Coated U700 Specimen Showing Localized Attack on Back Side Developed in 291.1 Hours at 1100°C in Burner Rig Cyclic Oxidation.	17
5.	Photomicrographs of Codep Coated U700 Specimen after 477.3 Hours at 1100°C in Burner Rig Cyclic Oxidation.	19
6.	Photomicrograph of a RT21 Coated René 80 Specimen Showing Localized Coating Failure after 98.5 hours at 1100° C in Burner Rig Cyclic Oxidation.	20
7.	Photomicrograph of RT21 and NiCoCrAlY Coatings after 600 Hours Isothermal Oxidation at 1100° C.	21
8.	Changes in Coil Inductance with Oxidation at 1100° C, Cyclic Exposure in the Burner Rig.	25
9.	Changes in Coil Inductance in Static Oxidation at 1100°C, Cyclic Exposure.	26
10a.	Rear View of a Burner Rig Specimens (a) after Task III Aging and (b) after 68.6 Hours of Task IV Hot Corrosion Test.	31
10b.	Rear View of Burner Rig Specimens (a) after Task III Aging and (b) after 60.0 Hours of Task IV Hot Corrosion Test.	32
11.	Task IV Results, 100 Hours of Preaging.	34
12.	Hot Corrosion Life Versus Isothermal Aging Time.	35
13.	Change in Coil Inductance with Hot Corrosion at 900° C of Aged Coated Specimens.	37
14.	Change in Coil Inductance with Hot Corrosion at 900° C of Aged RT21 Coated Specimens.	38

# LIST OF TABLES

<u>Table</u>		Page
1.	Test Information and Metallographic Measurements for Specimens Removed from Task II Burner Rig Hot Corrosion Test.	8
II.	Average Corrosion ( $\mu m$ ) of Uncoated Specimens from Task II Burner Rig Tests Based on Cross Section Area Measurements.	13
III.	Na <sub>2</sub> SO <sub>4</sub> Accumulation on Task II Burner Rig Test Specimens.	13
IV.	Electron Microprobe Analyses of Coatings before and after Various Aging Treatments at 1100° C.	23
٧.	Weight Changes and Coil Inductance Changes for Task III Specimens.	24
VI.	Specimens Removed from Task IV Burner Rig Test.	28
VII.	Metallographic Evaluations of Task III Control Specimens (1100° C Aging) and Visual Observations of Degradation in Subsequent Task IV Hot Corrosion Tests (900° C).	33
VIII.	Na <sub>2</sub> SO <sub>4</sub> Accumulation on Task IV Burner Rig Test Specimens.	39

#### SUMMARY

This is the second annual report of a 3-year program. The objectives of this program are to determine surface chemistry effects of oxide scale and coating composition on the hot corrosion life of selected alloys in the coated and uncoated condition, and to provide data for development of a hot corrosion life prediction model.

The coated and uncoated alloys are evaluated in two ways: by high velocity hot corrosion rig testing in the as-processed condition and after various aging treatments designed to simulate surface chemistry effects of in-service conditions. The alloys used are Udimet 700 and René 80, both uncoated and coated with RT21, Codep, or NiCoCrAlY. All test specimens in this program are periodically removed from test for inspection and for nondestructive tracking of the progress of corrosion by measurement of 10 MHz coil inductance changes by the technique developed at NASA-Lewis Research Center.

Later in the program, data from the evaluation of all rig-tested specimens will be used to develop a hot corrosion life prediction model and methodology for the specific alloys and coatings tested on the program. The capability of the life prediction model and methodology will then be tested by conducting additional hot corrosion tests on both the program coatings and alloys as well as other coatings and alloys.

The hot corrosion tests in both the as-processed and aged condition have been completed, and much of the posttest evaluation. Baseline coatings lives have been established, and for all three coatings substantial life decreases were observed after aging. The life decreases were dramatically greater for coatings on U700 than on René 80.

In addition to the laboratory test program, a comprehensive evaluation of six service-run turbine components was initiated to: (1) establish extent and mechanism of hot corrosion degradation and (2) correlate observations to engine operating history, where possible. This study has been completed and was summarized in the first annual report. Sulfidation was observed in five of the six components.

Qualitatively, the extent of corrosion appeared to be inversely proportional to average length of mission. However, with such a limited sampling it was not possible to determine whether this reflects fraction of time under takeoff conditions (higher temperatures), fraction of time near ground level (higher propensity for contamination), or some other related parameter.

#### INTRODUCTION

The program is a 36-month joint effort including the Engineering Materials Technology Laboratories of the General Electric Company Aircraft Engine Business Group and the Materials and Manufacturing Technology Center of TRW, Inc. It is divided into the following five tasks.

Task I involves the evaluation of six turbine components showing visual evidence of hot corrosion and having known operating history, to establish the degree of degradation and mechanisms in the corroded areas. Correlation of the corrosion with operating conditions is made to the extent possible. Evaluation includes chemical and X-ray diffraction analyses of surface scales/deposits as well as metallography, scanning electron microscopy, and electron microprobe examination. Both uncoated and coated hardware are included, and emphasis is placed on the cause and effect parameters that are associated with hot corrosion. The results of this study serve as a point of reference for the laboratory testing in the remainder of this program. All burner rig tests are performed by TRW Inc., Cleveland, Ohio.

Task II establishes a hot corrosion baseline for the program alloys and coatings in the as-processed condition and involves up to 1000 hours exposure of duplicate specimens in a Mach 0.3 burner rig using Jet A fuel (0.045-0.065% sulfur) and in which 0.5 ppm sodium by weight, as NaCl, is added to the combustion air. The test cycle is one hour at 900° C, followed by six minutes of forced air cooling to ambient temperature. Specimens are inspected at approximately 20 cycle intervals, and removed from test when evidence of hot corrosion is noted in three successive inspections. Additional specimens are also tested for time periods of 100, 300, and 500 hours if these time periods do not exceed 2/3 the time the original specimen of the same material was exposed. Specimens tested in this task include U700 and René 80, both uncoated and with the following coatings:

- Pack aluminide RT21 (Chromalloy) and Codep (General Electric)
- Low pressure plasma sprayed NiCoCrAlY (Ni-23Co-18Cr-12A1-0.3Y)

In Task III, triplicate specimens of the above coated alloys are aged at 1100°C under a variety of conditions, both oxidizing and inert environments, to induce coating-alloy interdiffusion with and without surface oxidation. These specimens are subsequently hot corrosion tested in Task IV under the same conditions as in Task II to determine the effects of aging on hot corrosion behavior.

The aging treatments in Task III are:

- Isothermal for 100 hours in a vacuum
- Isothermal air furnace oxidation for 100, 300, and 600 hours
- One hour air furnace cycles for 100 hours
- Cyclic burner rig oxidation for 100, 300 and 600 hours

One specimen representing each of the above conditions is evaluated using the methods outlined in Task I.

In Task IV, the remaining duplicate specimens representing each condition will be used, and an empirical hot corrosion life prediction model will be proposed. All data will be consolidated, including the effects of aging on hot corrosion behavior.

Task V will be a hot corrosion cyclic burner rig test designed to check the validity of the proposed life prediction model. Duplicate specimens of a maximum of four alloys and five coatings will be tested up to a maximum of 1000 hours and evaluated as in Tasks II and IV. The alloys and coatings will be those from this program plus possible additional alloy-coating systems selected by the NASA Project Manager.

The Task V experiment will be designed to test the following aspects of the life prediction model developed in Task IV.

- Using the same alloys and coatings tested in Tasks II and IV, burner rig tests will be performed under different test conditions (various salt levels) and with modifications of the aging conditions used in Task III.
- Other alloys and coatings may be included, depending on final evaluation of results from Tasks II and IV and the impact on the model.

#### TASK II - BASELINE HOT CORROSION TESTS

All hot corrosion testing in this program is being performed at TRW, Inc., Cleveland, Ohio, in high velocity (0.3 Mach) burner rigs operating at one atmosphere. Data have been obtained on uncoated and coated alloys to be compared later (Task IV) with data obtained under the same test conditions, and on the same materials, but with specimens previously subjected to a variety of aging conditions (Task III).

Materials for this Task II burner rig hot corrosion test include Udimet 700 and uncoated René 80 with the following coatings:

- RT21 aluminide (Chromalloy American Corp.)
- Codep aluminide (General Electric Co.)
- Ni23Col8Cr12Al0.3Y applied by vacuum (low pressure) plasma spraying Nominal major constituents of the substrate alloys in weight percent are:
- Udimet 700 14.5 Cr, 4.3 Al, 3.4 Ti, 15.0 Co, 4.2 Mo, balance Ni
- René 80 14.0 Cr, 3.0 Al, 5.0 Ti, 9.5 Co, 4.0 Mo, 4.0 W, balance Ni

Details concerning specimen preparation were given in the first Annual Report (Reference 1) which also includes photomicrographs and electron microprobe (EMP) analyses of cross-sections of all coating/substrate systems. Thicknesses of these coatings, including diffusion zone, were reported as:

Codep/René 80	50 μm
Codep/U700	84
RT21 René 80	81
RT21/U700	79
NiCoCrAlY/René 80	98
NiCoCrAlY/U700	98

Throughout this report, specimens are coded "X" for U700 and "L" for René 80 substrates.

Illustrations of the burner rig, nozzle, salt solution spray device and description of operating procedure have been presented previously (Reference 1). Burner rig tests were conducted under the conditions listed below. (Jet A fuel was used, with a sulfur content of 0.055%.)

- 1. Specimen temperature 900° C ± 9° C.
- 2. Test Cycle one hour at temperature followed by six minutes of forced air cooling.
- 3. Sodium Concentration 0.5 ppm sodium (±10%) in the combustion gases introduced as aqueous NaC1.
- 4. Combustion air preheat temperature 232° C ± 10° C.
- 5. Specimens eight positioned equally on a 4.2 cm (1.64 inch) diameter circle of a holder rotating at 600 rpm.
- 6. Burner nozzle throat diameter 2.54 cm (1.0 inch).
- 7. Burner pressure 1.0 psig.
- 8. Nozzle throat to nearest specimen 4.45 cm (1.75 inch).

The test plan included duplicate specimens randomly distributed between two burner rigs of U700 and René 80, both uncoated, and with RT21, Codep, or NiCoCrAlY coatings. Specimens were removed from test at approximately 20 cycle intervals for inspection (in practice about 18 to 24 cycles). When visual evidence of hot corrosion was apparent at three successive inspections, the specimen was removed. The visual appearance of all specimens was documented photographically at each inspection. The plan also included the testing of single specimens of each material for time periods of 100, 300, and 500 hours, if these time periods did not exceed 2/3 the time the original specimens of the same material were exposed. Thus, for a material that was not removed until at least 750 hours for visual reasons, the test provided evaluation of the extent of corrosion at four different time intervals.

Posttest evaluation of specimens was performed by the traditional destructive techniques used in Task I. In addition, this program provides an opportunity to extend the recent pioneering studies at NASA-Lewis Research Center in which the progress of hot corrosion (or oxidation) is monitored, nondestructively, by the measurement at each inspection interval of the inductance of a coil at high frequency with the test specimens being a core (Reference 2).

Under carefully controlled conditions, (noting that at high frequencies only the outer surface of the metallic core will influence the measurement) the coil inductance can respond either to a change in metallic core volume (metal converted to oxide scale) or to a change in composition of the metallic surface (formation of a depletion zone within the alloy as scale forms). In either event, results from such measurements afford high sensitivity in non-destructively following the changes that occur during environmental degradation. Results from this program shall contribute significantly to an understanding and interpretation of such measurements.

Coil inductance measurements were made with the burner rig specimen positioned in a coil (Reference 1, Figure 19) such that the center of the flame impingement zone, 2.5 cm from the top of the specimen, was in the center of the coil. Measurements were made in series mode with a multi-frequency LCR meter at a frequency of 10 MHz. At least two measurements were taken with each specimen core. For each measurement, the specimen was removed from the coil and replaced randomly to average the core-coil axial alignment.

#### Results

Testing has been completed for the full slate of 32 specimens. Of these, two of each coating/alloy combination were run to full term per the criteria described above, or to 1000 hours, whichever occurred first. Test information and metallographic measurements are summarized in Table I. Of the seven specimens that were in test for the full 1000 hours, the two that exhibited breach of coating did not in fact show visual evidence of hot corrosion. Of the other five not showing breach of coating, three had been visually judged as indicating hot corrosion. These discrepancies point up the dual problem in evaluation, particularly for coated specimens for which corrosion is frequently localized:

- It is difficult to capture localized pits in a single planar section. Conceivably, specimens which show visual evidence of hot corrosion have sites elsewhere where pitting has taken place down to the coating/substrate interface.
- Visual evidence of hot corrosion of a coated specimen is generally not apparent until the more rapidly corroding substrate at the site of a pit causes an eruption of scale.

Test Information and Metallographic Measurements for Specimens Removed from Task II Burner Rig Hot Corrosion Test.

Code∆	Alloy	Coating	Hours	Initial Coating Thickness, µm	Coil Inductance Change, µH	Maximum Corrosion Attack, µm
X3 X19 X108 L54 L77	U700 U700 U700 U,700 René 80 René 80		142.65 157.6 105.5 162.65 167.7		0.315 0.259 0.230 0.204 0.186	+ *
L99 X14 X8	U700 U700	Codep Codep	104.1 602.0 424.8	84 84	0.087 0.012 0.017	285* 40*
X22 X50 L3 L7 L12	U700 U700 René 80 René 80 René 80	Codep Codep Codep Codep Codep	298.9 98.9 527.8 438.9 296.6	84 84 50 50 50	0.007 -0.005 0.034 0.023 0.022	235* 30 250* 40* 130*
L110 X78 X66	René 80 U700 U700	Codep Codep RT21 RT21	103.0 1004.6 997.1	50 50 79 79	0.016 -0.005 0.000	25 95 10*
X104 X114 L36 L44	U700 U700 René 80 René 80	RT21 RT21 RT21 RT21	295.6 97.0 1004.6 697.1	79 79 81 81	-0.001 -0.004 0.016 0.012	15 10 20* 180*
L8 L106 X56 X30	René 80 René 80 U700 U700	RT21 RT21 NiCoCrAlY NiCoCrAlY	300.3 100.7 1004.6 997.1	81 81 98 98	-0.020 -0.017 0.005 0.010	10 10 70 100
X79 X65 X131	U700 U700 U700	NiCoCrAlY NiCoCrAlY NiCoCrAlY	470.8 298.9 138.9	98 98 98 98	0.003 0.002 0.006	10 10 10
L41 L9 L49 L128 L133	René 80 René 80 René 80 René 80 René 80	NiCoCrAlY NiCoCrAlY NiCoCrAlY NiCoCrAlY NiCoCrAlY	1004.6 997.1 528.1 152.7 100.7	98 98 98 98 98	0.002 0.004 0.006 0.005 0.002	35* 70 30 25 20

The first two entries for each materials system were tested to full term, that is, two 20 hour cycles after first visual evidence of corrosion (or 1000 hours). Other specimens were tested for fractional times.

See Table II.
Visual evidence of hot corrosion.

Note also that for several Codep coated and RT21 coated specimens, corrosion has proceeded considerably into the substrate alloy, (see Table I). This is a consequence, in part, of the criterion for removal of a specimen, namely that it be exposed to test for two more 20-cycle periods after visual evidence of corrosion is noted. For coated specimens, visual evidence nominally coincides with coating penetration.

These uncertainties, of course, apply to all the other coated specimens as well. Nevertheless, it is clear that the RT21 coated specimens on average performed better than the Codep coated specimens. Based on EMP analyses (Reference 1) and additional evaluation, no significant compositional differences exist between the two coatings and both have the structural features of inward aluminide coatings. On the other hand, the RT21 coating shows evidence of entrapment of small oxide particles in the outer 5  $\mu m$ , while the Codep coating does not. Whether this difference is the cause of differences in hot corrosion lives cannot be concluded with certainty; yet it may be noted in anticipation of results from Task IV testing that no consistent difference exists for specimens preaged at 1100° C.

Additional indication of a difference between these two coatings is displayed in Figure 1. Accumulative coil inductance changes for seven individual Codep coated and RT21 coated specimens, together with an average curve for four NiCoCrAlY coated specimens, are shown. The time scale below 200 hours is expanded to show clearly the negative changes that appeared at the first inspection for the aluminide coated specimens. These are considered real inasmuch as the NiCoCrAlY coated specimens showed a much smaller change; for these, the small changes as a function of time may well represent the normal scatter in the measurements. Note that all four of the Codep coated specimens show greater and faster increases which parallel the metallographic observations.

In all instances, Type 1 sulfidation was clearly present in the RT21 coated or Codep coated specimens, either within the coating where still present or in a  $\gamma'$  depleted region in the underlying alloy where the coating was breached. Typical microstructures are shown in Figure 2. X-ray oscillographs on several of these specimens showed the oxide scale to be aluminum rich, with lesser amounts of Cr and Ti not uniformly distributed.

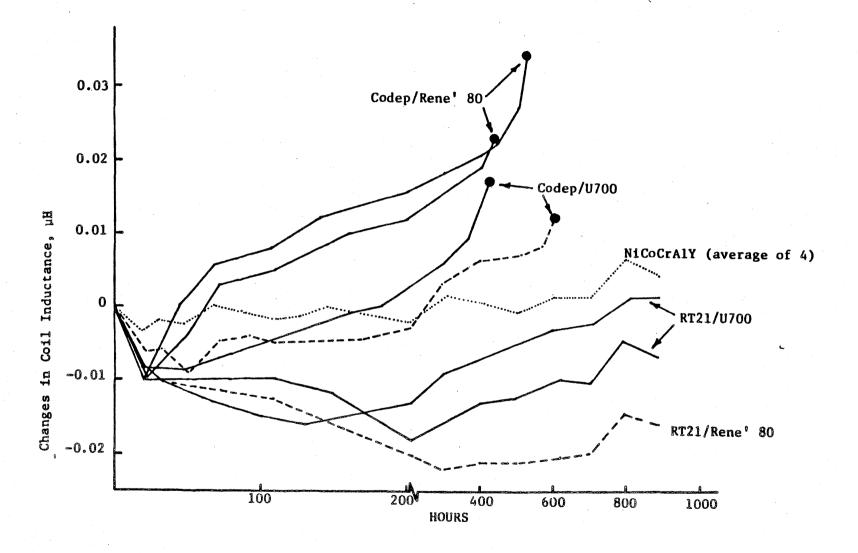
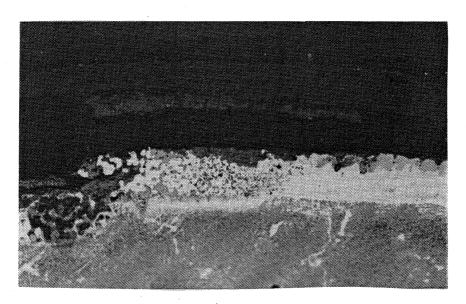
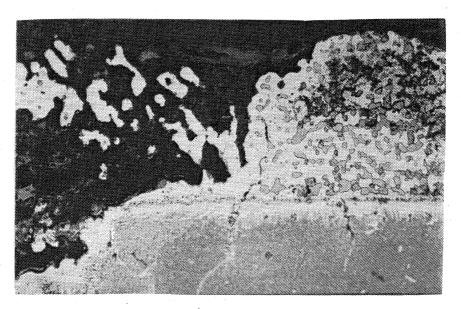


Figure 1. Changes in Coil Inductance with Hot Corrosion at 900° C, Cyclic Exposure.



RT21/René 80, 697.1 hours

(Etched, 200x)



Codep/U700, 602.0 hours

Figure 2. Photomicrographs from burner rig hot corrosion tests at  $900^{\circ}$  C. Type 1 sulfides are present.

The NiCoCrAlY coating is considerably more resistant to hot corrosion. Complete penetration was noted in only one of the four full term 1000 hour specimens. A series of three typical microstructures depicting the progress of hot corrosion with time is shown in Figure 3. The development of a substantial depleted layer at full term is shown, determined by EMP analysis to be primarily a loss of Cr, down to about half of the nominal 18%. There was a corresponding increase in Ni, but only a small decrease in Al. Type 1 sulfides could not be visually identified in the depletion zone as might have been expected, and this was confirmed with X-ray oscillographs. However, sulfur was found in local regions of scale deep within pits, primarily associated with Cr. Most of the scale was Al and Ti rich, and devoid of sulfur.

With respect to uncoated specimens, the corrosive attack is generally deeper than with coated specimens and is not localized; that is, corrosion occurs continuously around the entire circumference. Thus, the location of the original surface cannot be identified, which means that a reliable measurement of "maximum depth of penetration" is not possible. As an alternative, area measurements with the Leitz TAS-Plus Image Analyzer were used for these specimens. For each specimen, two measurements were made on etched mounts.

- Area of unaffected metal (base of the γ' depleted layer).
- Area at the metal/scale interface.

These measurements were converted to average radius values, and compared with pretest dimensions, (see Table II). Although these are average values representing nonuniform corrosion, comparison with coil inductance changes is appropriate. A real correlation is difficult for the following three reasons.

- Coil inductance changes reflect compositional changes in the outer layer of metal as well as cross-section changes.
- 2. Neither coil inductance changes nor corrosion are linear functions of time; a substantial fraction of the total coil inductance change occurs during the corrosion incubation period. (Reference 1, Figure 21).
- 3. The extent of corrosion observed metallographically is measured in a plane, while the coil inductance reflects total corrosion over the length of the specimen within the coil.

Table II. Average Corrosion ( $\mu m$ ) of Uncoated Specimens from Task II Burner Rig Tests Based on Cross Section Area Measurements.

Code	Alloy	Hours	Coil Inductance Change, µm	Metal Loss	Depletion Zone	Total Affected Metal
X19	U700	157.6	0.259	310	45	355
X21*	U700	147.7	0.270	360	80	440
X3	U700	142.65	0.315	265	100	365
X108	U700	105.5	0.230	100	50	150
L77	René 80	167.7	0.186	100	80	180
L54	René 80	162.65	0.204	110	90	200
L99	René 80	104.1	0.087	40	35	75

 $<sup>\</sup>star$  Dummy specimen, not in original test plan.

Table III.  ${\tt Na_2SO_4}$  Accumulation on Task II Burner Rig Test Specimens.

Specimen	Test Time,	Na <sub>2</sub> SO <sub>4</sub>							
Number	Hours	Na, mg	mg/cm <sup>2</sup>	mg/cm <sup>2</sup> 100 Hours					
L77 X19 L7 X8 X73 L36	167.7 157.6 438.9 424.8 1004.6	8.3 7.9 22.8 20.9 27.9 27.7	1.28 1.22 3.51 3.22 4.30 4.28	0.76 0.78 0.80 0.76 0.43					
L41	1004.6	23.9	3.69	0.37					

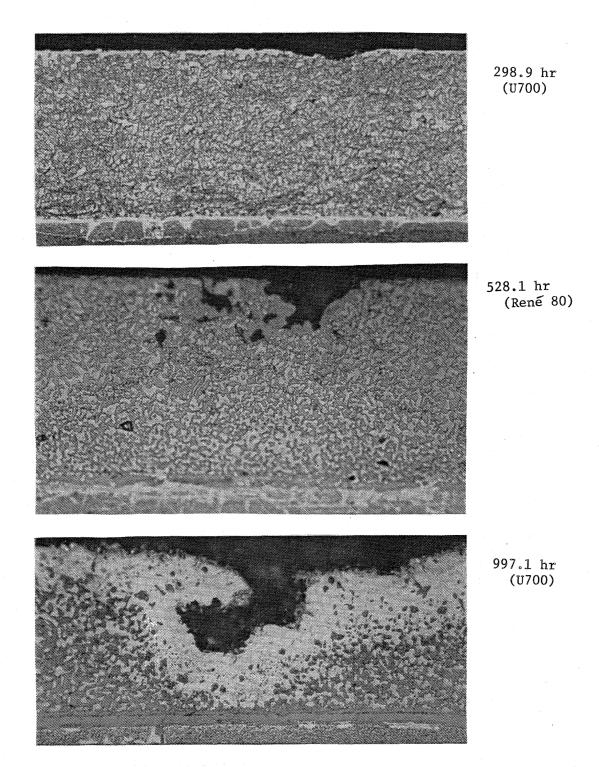


Figure 3. Photomicrographs of NiCoCrAlY coated specimens from hot corrosion burner rig test at 900° C showing progressive degradation with time.

(Etched, 500x)

The extent of corrosion increases with time, as expected, and René 80 is about twice as resistant to corrosion as U700, also predictable from previous extensive burner rig studies and field experience. The depletion zone in all specimens showed the expected Type 1 sulfidation, amply demonstrated with X-ray oscillographs. Scales were typically Al rich with lesser amounts of Ti and Cr. Typical microstructures were presented previously (Reference 1).

As further documentation of these burner rig tests, salt accumulation  $(Na_2SO_4)$  was measured on several specimens. Results are given in Table III. The rate of accumulation is essentially constant to 438.9 hours, but is less for the 1004 hour specimens. This may reflect mechanical instability of the salt deposit, that is, spallation, above a certain thickness. The alternate explanation that the salt flux was lower in the latter part of the test may be discarded since several short term dummy specimens exposed at various times after the initial 438 hour point exhibited deposition rates typical of the earlier specimens.

#### TASK III - AGING TREATMENTS

The purpose of this Task is to prepare specimens for subsequent hot corrosion testing (Task IV) in which the coating composition has been altered by heat treatment at 1100° C in either inert or oxidizing environments. This induces coating/substrate alloy interdiffusion with and without surface oxidation. Each treatment included triplicate specimens of all six coating/substrate systems tested in Task II. Conditions were:

* Cyclic, Static Air	100 hours
* Cyclic, Burner Rig	100, 300, 600 hours
Isothermal, Static Air	100, 300, 600 hours
Isothermal, Vacuum	100 hours

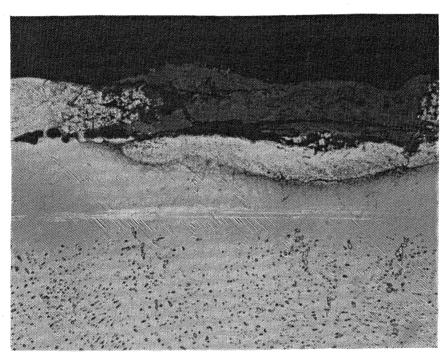
<sup>\*</sup> One hour cycles, inspections at 20 cycle intervals.

For the burner rig exposures, specimens were randomly distributed between four rigs. Weight and coil inductance measurements were made before and after heat treatment, and at intermediate inspections for cyclic exposures. One of each group of three was sectioned for metallographic measurement and the other two retained for hot corrosion testing in Task IV.

#### Results

There were two somewhat unexpected results:

- 1. The vacuum heat treatment caused substantial weight losses, suggesting volatilization. These losses are selective, confirmed by the appearance of a thin layer (5-10µm) in the sectioned NiCoCrAlY coated specimens, in which the Cr was down to 7% from the nominal 18%.
- 2. In the burner rig exposures, very localized pitting on the back side of the NiCoCrAlY coated specimens developed; consequently, those intended for 600 hours exposures were removed at times ranging from 352 to 420 hours when visual examination suggested coating penetration. A photomicrograph showing the localized features of this degradation is shown in Figure 4. Another specimen which showed coating penetration (U700-380 hours) was carefully examined for



200x

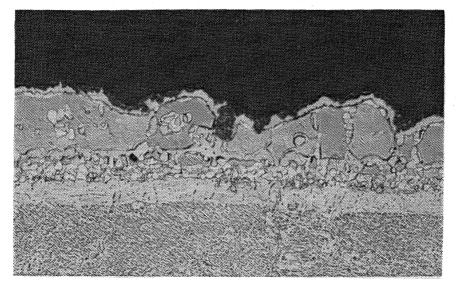
Figure 4. Photomicrograph of NiCoCrAlY coated U700 specimen showing localized attack on back side developed in 291.1 hours at  $1100\,^{\circ}\text{C}$  in burner rig cyclic oxidation.

sulfides which could have indicated salt contamination. None was found. Also, if the temperature were significantly higher on the back side, the Codep and RT21 coatings presumably would have failed locally; this did not happen. There is as yet no explanation why the NiCoCrAlY coatings exhibited such nonuniform oxidation in contrast to Codep and RT21. (NOTE: The NiCoCrAlY coated specimens exposed for the full 600 hours under static conditions showed a very thin coherent alumina scale but essentially no metal loss. (See Figure 7.)

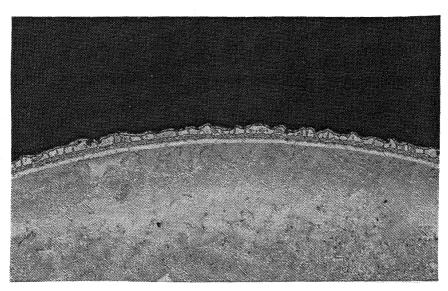
The Codep coated and RT21 coated specimens also showed visual evidence of coating penetration at far less than 600 hours in the burner rig, and those planned for the full exposure were removed at times ranging from 357 to 488 hours. However, the site of initial failure was at the back of the specimen in only a few instances.

In general, oxidation was fairly uniform around the circumference, rather scalloped, though somewhat more on the back side. Typical microstructures are shown in Figure 5. In one instance, local coating failure was confirmed in only 98.5 hours for a RT21 coated René 80 specimen (Figure 6). Although this failure occurred on the back side of the specimen, it was considerably removed from the 180° location at or near which all of the NiCoCrAlY coated specimens showed localized degradation.

Even for specimens aged in static air, there was evidence of occasional localized degradation for all three coatings. Specific observations and maximum penetration measurements for all sectioned specimens are deferred to the Task IV discussion since this information directly relates to coating lives in the subsequent hot corrosion test. Typical microstructures for the RT21 and NiCoCrAlY coatings after 600 hours isothermal oxidation at 1100° C are shown in Figure 7 for typical regions. For the RT21 coating, the scattered light phase at the outer surface is  $\gamma'$  resulting from a decrease in Al content. In the diffusion zone, separation of carbides and/or intermetallics has occurred. These changes are visible but much less so after 100 and 300 hours. The voids shown at the NiCoCrAlY/ substrate interface are typical of all the aged specimens with this coating.



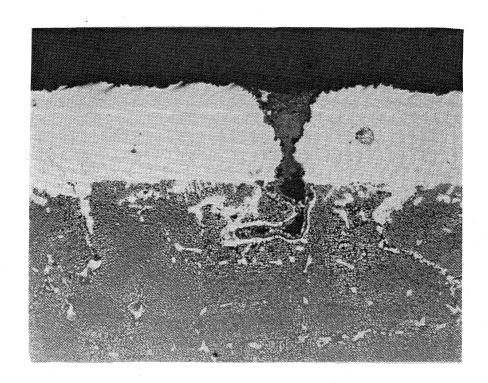
200x



32x

Figure 5. Photomicrographs of a Codep coated U700 specimen after 477.3 hours at  $1100^{\circ}$ C in burner rig cyclic oxidation.

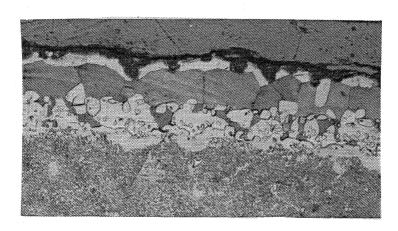
Note local irregularities yet generally uniform degradation.

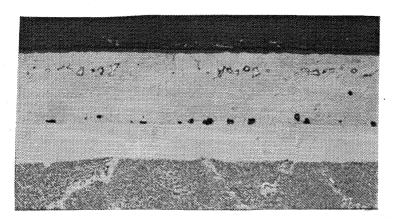


(Etched, 200x)

Figure 6. Photomicrograph of a RT21 coated René 80 specimen showing localized coating failure after 98.5 hours at 1100 °C in burner rig cyclic oxidation.

RT21/René 80





NiCoCrAlY/René 80

(Etched, 200x)

Figure 7. Photomicrographs of RT21 and NiCoCrAlY coatings after 600 hours isothermal oxidation at  $1100\,^{\circ}\text{C}$ .

Also of use in Task IV are the results of electron microprobe (EMP) examination. Selected quantitative analyses are summarized in Table IV together with X-ray oscillograph observations on several scales. Included for comparison are results obtained previously for as-fabricated specimens. All of these values are averages of several analyses at different depths within the bulk coating, that is, neglecting the diffusion zone and near the outer surface. (For three NiCoCrAlY coated specimens, analyses for Cr and Al just beneath the scale are included.) Compositional changes are substantial, notably caused by inward diffusion of Al and outward diffusion of substrate elements. Of particular interest is the similarity for air and vacuum exposures; composition changes from interdiffusion dominate over those resulting from oxidation.

In addition to the above metallographic and EMP observations, weight changes and coil inductance changes were recorded. These are summarized in Table V. The latter for cyclic exposures both in the burner rig and under static conditions are shown in Figures 8 and 9. There were no negative changes at the first inspection as previously noted for coated specimens in the Task II hot corrosion test at 900° C (Figure 1).

Further comments are required with respect to the weight changes in Table V. Since Codep and RT21 are pack cementation coatings, they covered the entire specimen surface. For these, weight changes were progressively positive with time and did not suggest scale spallation except for the longest burner rig exposures for which localized blistering (and probably spalling) occurred, as discussed above.

On the other hand, the NiCoCrAlY coating was plasma sprayed, leaving the bottom end of the specimens uncoated. These uncoated ends were given some protection with a slurry aluminide coating to prevent gross degradation in the long term aging treatments. This protection was adequate, yet only marginal as indicated by the significant weight losses for several of these specimens when aged for 300 hours or more.

Table IV - Electron Microprobe Analyses of Coatings Before and After Various Aging Treatments at 1100° C.

### Weight Percent

Outer Layer

				Bulk Coating					of Coating				
Code	Alloy	Coating	Aging Conditions	Ti	Cr	Со	Ni	W	Al	Мо	Al	Cr	Scale
L96	René 80	Codep	None	0.7	5.0	6.5	52.0	0.9	32.0	0.8			
L101	René 80	RT21	None	0.4	3.5	6.0	52.0	0.0	32.0	0.4			
L103	René 80	·RT21	100 Hour Vacuum	4.0	7.5	9.3	61.0	0.1	18.0	0.3	1		
L94	René 80	Codep	100 Hour Vacuum	3.7	6.5	9.4	64.0	0.1	17.0	0.4			
L125	René 80	Codep	100 Hour Cyclic	3.6	6.3	8.5	63.0	0.5	17.0	0.3			(1)
X26	U700	Codep	None	0.8	3.8	10.6	52.0		32.0	0.6			
X113	บ700	RT21	None	0.5	3.0	10.4	50.0		31.0	0.3			
X123	บ700	RT21	100 Hour Vacuum	3.1	6.7	12.4	61.0		17.0	0.6			
X130	U700	Codep	100 Hour Cyclic	2.5	6.7	11.0	61.0		18.0	0.5			(1)
X87	U700	RT21	98.5 Hour Burner Rig	3.0	7.7	11.8	60.0		17.0	1.0			
L88	René 80	NiCoCrAlY	None	0.2	20.1	18.8	43.0		13.0				
L111	René 80	NiCoCrAlY	100 Hour Vacuum	1.7	14.4	18.7	53.0	0.4	11.0	0.7	17	7	
L37	René 80	NiCoCrAlY	100 Hour Cyclic	1.8	15.4	17.4	55.0	0.5	12.0	0.0			(2)
L57	René 80	NiCoCrAlY	600 Hour Isothermal	2.5	18.8	16.1	54.0	1.2	7.8	1.3	1		(2)
L5	René 80	NiCoCrAlY	98.5 Hour Burner Rig	1.8	16.2	17.2	54.0	0.6	10.7	1.2	9	21	(2)
X39	บ700	NiCoCrAlY	None	0.4	20.0	20.9	44.0		12.0				
X34	U700	NiCoCrAlY	100 Hour Cylic	1.4	15.6	19.4	54.0		14.0	0.2			(2)
X48	U700	NiCoCrAlY	600 Hour Isothermal	2.2	16.7	18.6	54.0		7.5	1.9			(2)
X13	U700	NiCoCrAlY	98.5 Hour Burner Rig	1.8	15.1	18.6	54.0		11.6	0.4	9	18	(2)
X15	U700	NiCoCrAlY	291.1 Hour Burner Rig	i	17.1	18.9	55.0		7.4	1.9			(3)

<sup>(1)</sup> Coherent  ${\rm Al}_2{\rm O}_3$  scale with scattered Ti, especially at pits. (2) Coherent  ${\rm Al}_2{\rm O}_3$ , no Ti. (3) In pit, high Al, Ti with scattered Cr, Y.

Table V. - Weight Changes and Coil Inductance Changes for Task III Specimens.

		100 Ho	urs	300 Ho	urs	600 Hours		
Material	Test Condition	mg	μΉ	mg	μΉ	mg	μH	
Codep/U700	One hour cyclic	12 (98.5)(2)	0.0200	16 (306.1)	0.0280	12 (477.3)	0.0265	
RT21/U700	burner rig (1)	12 (98.5)	0.0192	12 (306.1)	0.0347	5 (482.1)	0.0275	
NiCoCrAlY/U700		12 (98.5)	0.0042	-17 (291.1)	0.0116	2 (380.0)	0.0106	
Codep/René 80		13 (98.5)	0.0246	14 (306.1)	0.0264	1 (357.1)	0.0316	
RT21/René 80		12 (98.5)	0.0106	14 (306.1)	0.0197	7 (482.1)	0.0216	
NiCoCrAlY/René 80		14 (98.5)	0.0084	12 (291.1)	0.0086	16 (420.0)	0.0093	
Codep/U700	Isothermal	9.	0.0174	36	0.0227	37	0.0290	
RT21/U700	furnace oxidation	8	0.0173	27	0.0204	27	0.0310	
NiCoCrAlY/U700	(3)	10	0.0017	7	0.0056	21	0.0096	
Codep/René 80		16	0.0212	25	0.0266	27	0.0347	
RT21/René 80		10	0.0059	25	0.0161	19	0.0241	
NiCoCrAlY/René 80		14	0.0021	-27	0.0064	-93	0.0107	
Codep/U700	One hour cyclic	15	0.0229					
RT21/U700	furnace oxidation	13	0.0225					
NiCoCrAlY/U700	(3)	9	0.0058					
Codep/René 80	· ·	26	0.0264					
RT21/René 80		15	0.0236			· .		
NiCoCrAlY/René 80		-2	0.0059					
Codep/U700	Isothermal	-41	0.0190					
RT21/U700	vacuum (3)	-57	0.0170					
NiCoCrAlY/U700		-84	0.0017					
Codep/René 80		-107	0.0211					
RT21/René 80		-89	0.0154					
NiCoCrAlY/René 80		-105	0.0051					

<sup>(1)</sup> Results for single specimens, selected for metallographic evaluation.(2) ( ) = Actual test hours.

<sup>(3)</sup> Average results for triplicate specimens.

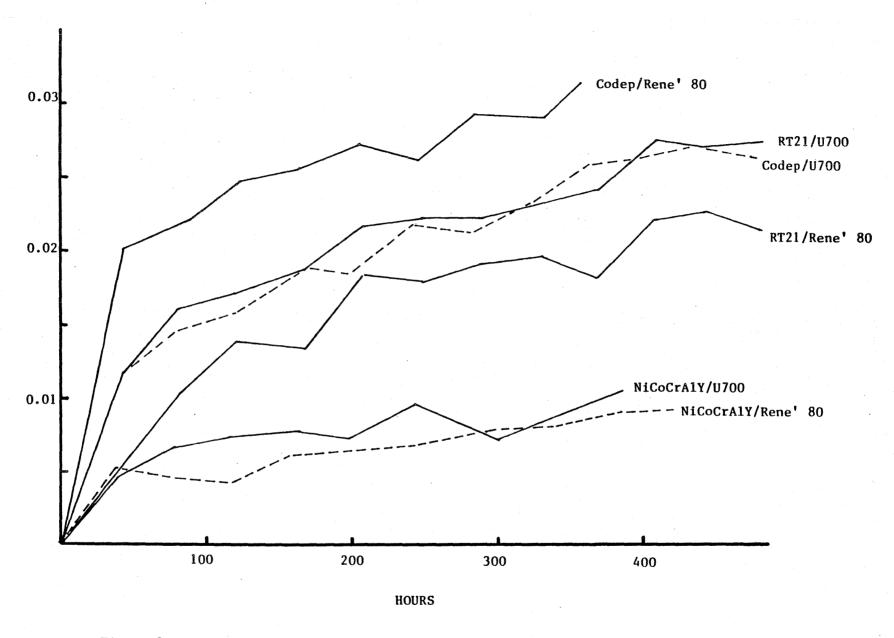


Figure 8. Changes in Coil Inductance with Oxidation at 1100° C, Cyclic Exposure in the Burner Rig.

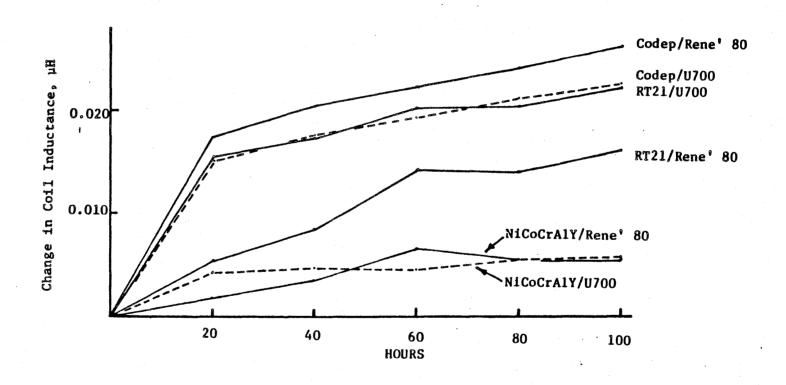


Figure 9. Changes in Coil Inductance in Static Oxidation at 1100° C, Cyclic Exposure.

## TASK IV - HOT CORROSION TESTS OF AGED SPECIMENS

This hot corrosion test included a total of 96 specimens, duplicates of six coating/alloy systems representing eight aging conditions. The specimens were randomly distributed between four burner rigs. All specimens were exposed to coating failure or 1000 hours, whichever occurred first. As in Task II, specimens were removed after the first visual evidence of hot corrosion plus two additional twenty hour cyclic exposures. Test conditions were the same as in Task II.

All testing has been completed and sufficient metallographic evaluation has been performed to confirm that:

- Type 1 hot corrosion was consistently produced
- Visual judgement regarding coating failure is generally reliable.

Table VI includes aging history, hot corrosion test time, and coil inductance changes separately for the aging and hot corrosion exposures. Confirmation of coating integrity for the five NiCoCrAlY specimens marked "not failed" after the full 1000 hours of testing was obtained metallographically; localized corrosion to a depth of less than half the initial coating thickness of 98  $\mu m$  was observed.

Further metallographic evaluation and EMP analyses will provide a more detailed interpretation of these results. However, several general comments may be made.

For the ≥300 hour burner rig and 600 hour isothermal aging, most of the specimens exhibited a coating hot corrosion life of less than 100 hours. Noting that the criterion for specimen removal was exposure for two 20-hour cycles after first visual evidence of corrosion, it is clear that the coating was essentially failed at the beginning of the hot corrosion test. Thus, these results are of minimal use in evaluating the effect of aging on hot corrosion life. This comment is particularly valid for NiCoCrAlY coated specimens aged in the burner rig which, as discussed previously, exhibited extreme localized degradation on the back side away from the flame. For these, hot corrosion life was shorter than for the RT21 coated and Codep coated specimens.

Table VI. Specimens Removed From Task IV Burner Rig Test.

					T		
					1	k IV	
Code	Alloy	Coating	Task III Exposure	Change in Coil Inductance, µH	Rig No.	Hours	Change in Coil Inductance, µH
X-119	U-700	Codep	100 Hours Vacuum	0.0179	1	119.2	0.0233
X-103	U-700	Codep	100 Hours Vacuum	0.0220	2	140.2	0.0124
X-126	U-700	RT21	100 Hours Vacuum	0.0181	3	140.4	0.0108
X-90	บ-700	RT21	100 Hours Vacuum	0.0102	4	140.2	0.0177
X-74	· U-700	NiCoCrAlY	100 Hours Vacuum	0.0026	i	996.7*	0.0178
X-60	U-700	NiCoCrAlY	100 Hours Vacuum	0.0003	2	893.5	0.0277
L-71	René 80	Codep	100 Hours Vacuum	0.0244	3	200.4	0.0540
L-100	René 80	Codep	100 Hours Vacuum	0.0174	4	200.2	0.0289
L-108	René 80	RT21	100 Hours Vacuum	0.0183	1	353.2	0.0145
L-117	René 80	RT21	100 Hours Vacuum	0.0189	2	564.2	0.0452
L-104	René 80	NiCoCrAlY	100 Hours Vacuum	0.0055	1	996.7*	0.0110
L-92	René 80	NiCoCrAlY	100 Hours Vacuum	0.0047	2	999.6*	0.0107
X-105	บ-700	Codep	100 Hours Cyclic	0.0226	1	200.2	0.0188
X-71	U-700	Codep	100 Hours Cyclic	0.0225	3	200.2	0.0063
X-127	U-700	RT21	100 Hours Cyclic	0.0233	2	283.2	0.0083
X-134	U-700	RT21	100 Hours Cyclic	0.0227	4	200.4	0.0180
X-35	U-700	NiCoCrAlY	100 Hours Cyclic	i .	3		
X-37	U-700	NiCoCrAlY	100 Hours Cyclic	0.0048	4	524.4 605.6	0.0342
T 100	D	C- 4	100 03/-	0.0060			
L-102	René 80	Codep	100 Hours Cyclic	0.0260	2	765.5	0.0392
L-105	René 80	Codep	100 Hours Cyclic	0.0258	4	524.2	0.0533
L-109	René 80	RT21	100 Hours Cyclic	0.0115	1	556.4	0.0204
L-127	René 80	RT21	100 Hours Cyclic	0.0180	3	483.4	0.0578
L-42	René 80	NiCoCrAlY	100 Hours Cyclic	0.0077	3	1002.6	0.0710
L-45	René 80	NiCoCrAlY	100 Hours Cyclic	0.0038	4	920.2	0.0740
X-32	บ-700	Codep	100 Hours Isothermal	0.0174	1	140.0	0.0051
X-54	U-700	Codep	100 Hours Isothermal	0.0192	2	88.0	0.0295
X-81	U-700	RT21	100 Hours Isothermal	0.0174	3	100.0	0.0315
X-86	U-700	RT21	100 Hours Isothermal	0.0162	4	121.4	0.0365
X-53	U-700	NiCoCrAlY	100 Hours Isothermal	0.0022	1	179.2	0.0218
X-59	บ-700	NiCoCrAlY	100 Hours Isothermal	0.0023	2	999.6	0.0169
L-21	René 80	Codep	100 Hours Isothermal	0.0203	3	525.3	0.0433
L-25	René 80	Codep	100 Hours Isothermal	0.0226	4	505.4	0.0352
L-59	René 80	RT21	100 Hours Isothermal	0.0047	ı	203.4	0.0115
L-74	René 80	RT21	100 Hours Isothermal	0.0070	2	189.5	0.0169
L-63	René 80	NiCoCrAlY	100 Hours Isothermal	0.0018	3	1002.6*	0.0161
L-72	René 80	NiCoCrAlY	100 Hours Isothermal	0.0021	4	1001.4*	0.0177
X-122	บ-700	Codep	300 Hours Isothermal	0.0246	1	80.2	0.1229
X-98	U-700	Codep	300 Hours Isothermal	0.0214	2	242.2	0.0302
X-100	U-700	RT21	300 Hours Isothermal	0.0214	1	80.2	0.0453
X-111	U-700	RT21	300 Hours Isothermal	0.0220	2	121.0	0.0101
X-111	U-700	NiCoCrAlY	300 Hours Isothermal	0.0209	3	384.4	0.0101
X-84	U-700	NiCoCrAIY	300 Hours Isothermal	0.0053	4	384.0	0.0804
L-87	René 80	Codep	300 Hours Isothermal	0.0016	١,	256 2	0.0650
		•		0.0216	1 1	356.2	0.0658
L-70	René 80	Codep	300 Hours Isothermal	0.0286	2	334.1	0.1131
L-124	René 80	RT21	300 Hours Isothermal	0.0177	1	161.4	0.0150
L-85	René 80	RT21	300 Hours Isothermal	0.0189	2	233.1	0.0537
L-90	René 80	NiCoCrAlY	300 Hours Isothermal	0.0052	1	755.5	0.0459
L-89	René 80	NiCoCrAlY	300 Hours Isothermal	0.0077	2	625.3	0.0469

<sup>\*</sup>Coating not failed.

Table VI. Specimens Removed From Task IV Burner Rig Test. (Concluded)

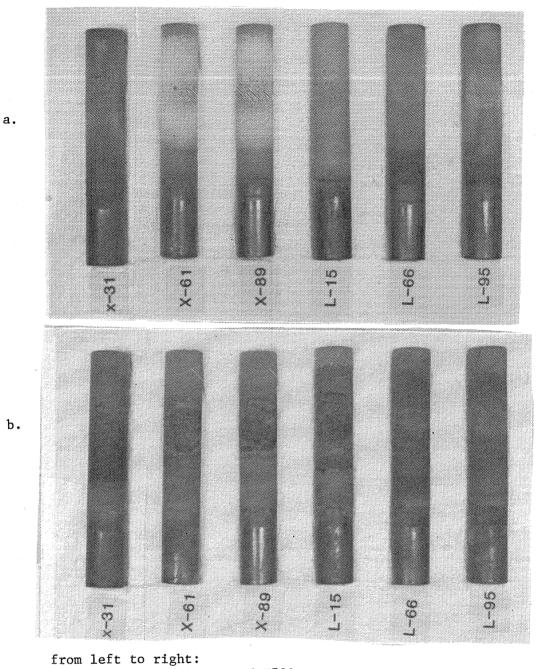
					Ta	sk IV	
				Change in Coil	Rig		Change in Coil
Code	Alloy	Coating		Inductance, µH	No.	Hours	Inductance, µH
X-18	Ŭ-700	Codep	600 Hours Isothermal	0.0314	1	80.2	0.1468
X-20	บ-700	Codep	600 Hours Isothermal	0.0317	2	80.0	0.1383
X-67	บ-700	RT21	600 Hours Isothermal	0.0329	1	72.0	0.0844
X-80	U-700	RT21	600 Hours Isothermal	0.0304	2	85.1	0.1478
X-52	บ-700	NiCoCrAlY	600 Hours Isothermal	0.0078	1	183.0	0.1475
X-47	U-700	NiCoCrAlY	600 Hours Isothermal	0.0084	2	181.3	0.2183
L-16	René 80	Códep	600 Hours Isothermal	0.0339	1	72.0	0.1077
L-19	René 80	Codep	600 Hours Isothermal	0.0355	2	60.0	0.1194
L-20	René 80	RT21	600 Hours Isothermal	0.0244	1	130.0	0.0514
L-10	René 80	RT21	600 Hours Isothermal	0.0228	2	120.1	0.0808
L-56	René 80	NiCoCrAlY	600 Hours Isothermal	0.0118	3	445.0	0.1312
L-60	René 80	NiCoCrAlY	600 Hours Isothermal	0.0096	4	525.4	0.1430
X-23	U-700	Codep	103 Hours Burner Rig	0.0213	1	115.0	0.0090
X-36	U-700	Codep	107 Hours Burner Rig	0.0240	3	121.0	0.0650
X-69	U-700	RT21	103 Hours Burner Rig	0.0207	2	112.1	0.0226
X-85	U-700	RT21	107 Hours Burner Rig	0.0194	4	140.0	0.0548
X-11	U-700	NiCoCrAlY	103 Hours Burner Rig	0.0079	1	179.2	0.0327
X-12	U-700	NiCoCrAlY	107 Hours Burner Rig	0.0068	2	825.5	0.0666
L-33	René 80	Codep	103 Hours Burner Rig	0.0238	2	278.5	0.0629
L-52	René 80	Codep	107 Hours Burner Rig	0.0276	4	159.4	0.0352
L-22	René 80	RT21	103 Hours Burner Rig	0.0140	1	377.2	0.0360
L-27	René 80	RT21	107 Hours Burner Rig	0.0143	3	221.3	0.0468
L-1	René 80	NiCoCrAlY	103 Hours Burner Rig	0.0082	3	766.5	0.0811
L-2	René 80	NiCoCrAlY	107 Hours Burner Rig	0.0046	4	745.6	0.0687
X-45	U-700	Codep	304 Hours Burner Rig	0.0281	3	75.1	0.1214
X-51	บ-700	Codep	298.2 Hours Burner Rig	0.0302	3	75.1	0.0836
X-95	U-700	RT21	304 Hours Burner Rig	0.0231	3	75.1	0.0737
X-96	U-700	RT21	298.2 Hours Burner Rig	0.0223	4	81.4	0.0323
X-24	U-700	NiCoCrAlY	289 Hours Burner Rig	0.0077	3	60.0	0.0937
X-28	U-700	NiCoCrAlY	293.2 Hours Burner Rig	0.0037	3	60.0	0.1011
L-66	René 80	Codep	304 Hours Burner Rig	0.0312	4	150.0	0.0456
L-69	René 80	Codep	298.2 Hours Burner Rig	0.0282	4	143.3	0.0641
L-39	René 80	RT21	304 Hours Burner Rig	0.0182	3	95.1	0.0563
L-50	René 80	RT21	298.2 Hours Burner Rig	0.0174	3	115.1	0.0583
L-13	René 80	NiCoCrAlY	289 Hours Burner Rig	0.0083	3	60.0	0.0268
L-15	René 80	NiCoCrAlY	293.2 Hours Burner Rig	0.0048	4	68.6	0.0492
X-61	บ-700	Codep	480.4 Hours Burner Rig	0.0383	4	68.6	0.0627
X-89	U-700	Codep	488.0 Hours Burner Rig	1	4	68.6	0.0667
X-101	บ-700	RT21	485.0 Hours Burner Rig		4	81.4	0.0782
X-118	U-700	RT21	477 Hours Burner Rig	0.0288	4	81.4	0.0935
X-29	U-700	NiCoCrAlY	374.6 Hours Burner Rig		3	60.0	0.1041
X-31	U-700	NiCoCrAlY	381 Hours Burner Rig	0.0114	4	68.6	0.1214
L-95	René 80	Codep	477 Hours Burner Rig	0.0515	4	108.9	0.0814
L-98	René 80	Codep	382.9 Hours Burner Rig		3	115.1	0.1100
L-51	René 80	RT21	485.0 Hours Burner Rig		4	101.4	0.0388
L-55	René 80	RT21	477 Hours Burner Rig	0.0205	4	101.4	0.0150
L-18	René 80	NiCoCrAlY	352.6 Hours Burner Rig		3	60.0	0.0441
L-24	René 80	NiCoCrAlY	415 Hours Burner Rig	0.0108	3	60.0	0.0424
l	1						

In most other instances, coating lives for duplicate specimens were in good agreement. For those few examples where this was not true, possible differences between burner rigs in operating parameters have been ruled out. Rather, this is believed to be largely caused by the relatively nonuniform degradation in the aging treatment. Typical specimen photographs are shown in Figures 10a and 10b.

Table VII shows metallographic observations from the aging treatments vis-a-vis visual observations from the subsequent hot corrosion test. The location of hot corrosion coating failures does not necessarily correspond to the location of coating "weak spots," developed in the prior aging; the two specimens of each coating/alloy system tested in hot corrosion may be either more or less degraded, and in different locations, than the Task III control specimen.

In spite of the above uncertainties, several observations may be made (see Figures 11 and 12). In these, averages for duplicate specimens are shown, except for two instances in Figure 11 for which widely disparate coatings lives were observed. (U700/NiCoCrAlY, isothermal and burner rig preoxidation).

- 1. For most of the aging conditions, the Codep and RT21 coatings exhibit considerably lower lives than observed in Task II. This reflects compositional changes, that is, a lower Al content, caused by the substantial interdiffusion in the 1100° C aging treatment in vacuum (see Table IV). The shorter lives for those specimens aged under cyclic oxidation conditions may also include the effect of a measurable loss in coating thickness during aging. However, the comparable removal times for specimens aged in vacuum versus air suggests that the compositional change is the overriding factor in decreasing the coating life.
- 2. On average, the lives of the RT21 and Codep coatings are in the same range, in contrast to the substantial differences observed in Task II. Whatever minor differences exist in these coatings in the as-processed condition are apparently erased by heat treatment at 1100° C, either inert or oxidizing environments.
- 3. For NiCoCrAlY coated specimens, there are parallel but less extreme decreases in hot corrosion life caused by the 1100° C aging. As shown in Table IV there were decreases in coating Al content at 1100° C but less dramatic and slower than in the Codep and RT21 coatings.
- 4. The most striking observation is that for all three coatings the corrosion life decrease, relative to Task II results, is far greater



From left to right:

NiCoCrAlY coated U700

Codep " U700

Codep " U700

NiCoCrAlY " René 80

Codep " René 80

Codep " René 80

Figure 10a. Rear view of burner rig specimens (a) after Task III aging and (b) after 68.6 hours of Task IV hot corrosion test.

a. 87-x

from left to right:

NiCoCrAlY coated U700
" " U700
" " U700
" René 80
" " René 80
" René 80

Figure 10b. Rear view of burner rig specimens (a) after Task III aging and (b) after 60.0 hours of Task IV hot corrosion test.

Table VII. Metallographic Evaluations of Task III Control Specimens (1100° C Aging) and Visual Observations of Degradation in Subsequent Task IV Hot Corrosion Tests (900° C).

	U700		René 80	
Task III Conditions	Task III Degradation, µm	Task IV Visual Observations (1)	Task III Degradation, µm	Task IV Visual Observations (1)
100 Hour Vacuum				
Codep RT21 NiCoCrAlY	nil nil nil	d a a,c	nil nil nil	d d a
100 Hour Cyclic	,			
Codep RT21 NiCoCrAlY	15 50 (local) nil	c d d	25 50 (local) nil	c,d d b,d
100 Hour Isothermal				
Codep RT21 NiCoCrAlY	nil nil 10 (local)	b b,d a,d	25 20 30 (local)	b d a
300 Hour Isothermal				
Codep RT21 NiCoCrAlY	35 (local) 20 nil	c c d	50 25 30 (local)	b b a,b
600 Hour Isothermal				
Codep RT21 NiCoCrAlY	35 (few) 35 (few) nil	b,c c b	251 35 (few) nil	c b d
100 Hour Burner Rig				
Codep RT21 NiCoCrAlY	70 (local) nil 20 (back)	c c d	50 80 35 (local)	b b b
300 Hour Burner Rig	·			
Codep RT21 NiCoCrAlY	45 40 100 (back)	c b a	20 45 70 (back)	d b b
>300 Hour Burner Rig			•	
Codep RT21 NiCoCrAlY	50 50 100 (back)	b c b	50 25 50 (back)	a,b b a,b

<sup>(1)</sup> Entries in this column are coded as follows:

a - Relatively local attack on back of specimen.

b - General attack on back half of specimen.

c - General attack, essentially all around.d - Relatively local attack in second and/or third quadrants from front (not in back).

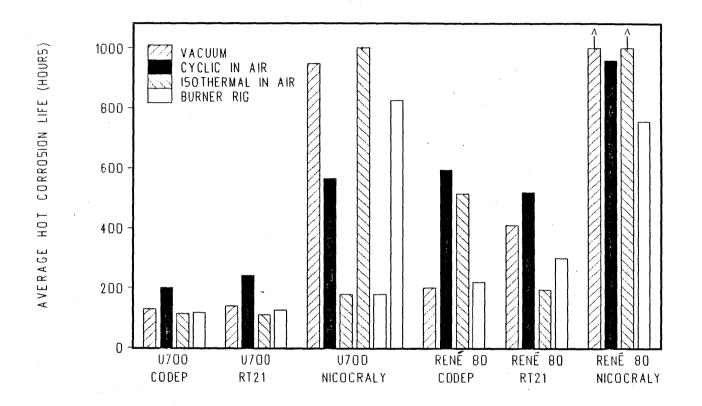


Figure 11. Task IV Results, 100 Hours of Preaging.

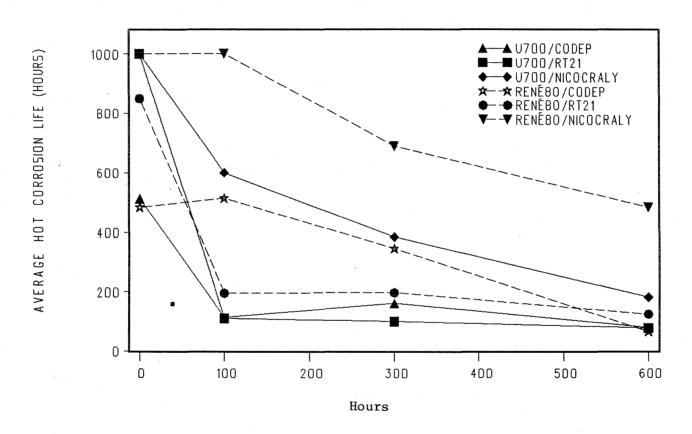


Figure 12. Hot Corrosion Life Versus Isothermal Aging Time.

for U700 substrates than for René 80 substrates. So far, no explanation has emerged. The EMP results in Table IV do not show any major differences in bulk coating compositions on the two substrate systems, although U700 and René 80 differ appreciably in levels of several minor elements. This may lead to significant differences between the two systems in the amount and composition of various carbide and intermetallic phases which develop during aging at 1100° C.

5. The results shown in Figure 12 demonstrate that for some coating/ substrate systems coating lives degrade progressively from 100 to 600 hours preoxidation; while for other systems maximum degradation has occurred in 100 hours preoxidation. This information will be useful in life prediction modeling and design of the Task V burner rig experiment.

In addition to these observations based on the information in Table VI and preliminary metallographic evaluations, selected coil inductance changes are shown in Figures 13 and 14. The offset at zero time indicates the change that occurred during the prior aging treatment.

The data shown in Figure 13 further reinforce the observation that both the RT21 and the NiCoCrAlY coatings exhibit a much shorter life on U700 than on René 80. The same is true for Codep coated specimens. (See Table VI.) For the RT21 coated specimens, the magnitude of the coil inductance changes is much greater than for their analogs tested in Task II in the as-fabricated condition (see Figure 1). This could indicate degradation of a greater fraction of the surface of aged specimens.

The small changes shown in Figure 14 do not suggest a consistent difference between vacuum and oxidizing aging treatments. This reinforces a comment made earlier, concerning coating lives, that compositional changes caused by coating/substrate interdiffusion are more important in decreasing coating life than are compositional changes involved in oxide scale formation.

In addition, salt accumulation (Na<sub>2</sub>SO<sub>4</sub>) was measured on twelve specimens. Results are given Table VIII. These accumulation rates are higher and more variable than observed in Task II (Table III) and may be a consequence of rougher specimen surfaces formed in the prior 1100° C aging. Thorough checking of burner rig operating conditions did not reveal any discrepancies. The higher salt accumulation rates may contribute to higher corrosion rates. However, such an effect is not considered a major one since coating lives of duplicate specimens (Table VI) were generally in agreement.

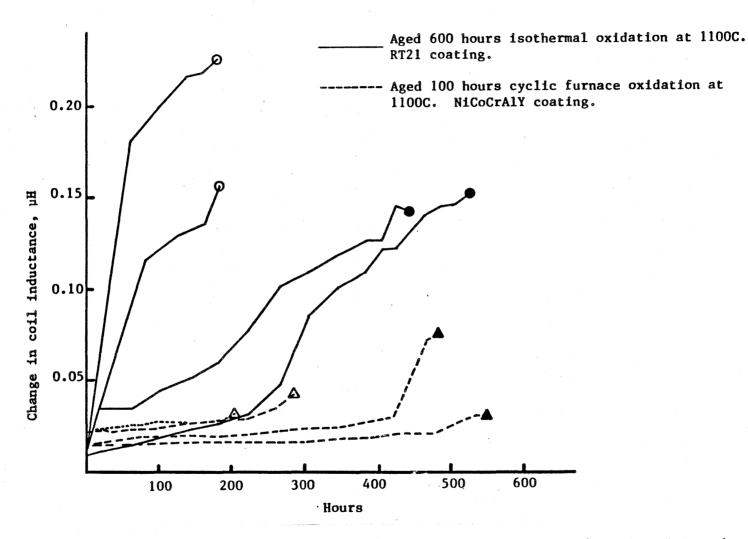


Figure 13. Change in Coil Inductance with Hot Corrosion at 900° C of Aged Coated Specimens. △OU700, ▲●René 80.

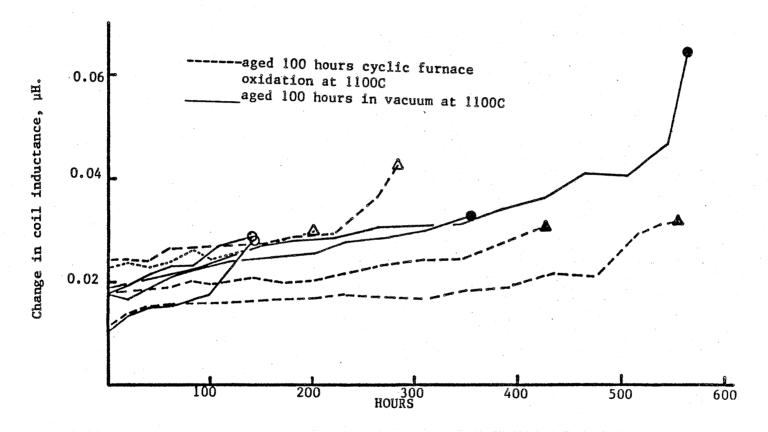


Figure 14. Change in Coil Inductance with Hot Corrosion at 900° C of Aged RT21 Coated Specimens. △OU700, ▲●René 80.

Table VIII.  ${\rm Na_2SO_4}$  Accumulation on Task IV Burner Rig Test Specimens.

Specimen Number	Test Time, Hours	Na, mg	mg/cm <sup>2</sup>	Na <sub>2</sub> SO <sub>4</sub> mg/cm <sup>2</sup> 100 hours
L71	200.4	24.0	3.71	1.85
L100	200.2	34.4	5.30	2.65
X103	140.2	39.0	6.02	4.29
X119	119.2	25.3	3.90	3.27
L59	203.4	12.3	1.90	0.93
X47	181.3	11.2	1.73	0.95
L33	278.5	27.1	4.18	1.50
L69	143.3	13.1	2.02	1.41
L50	115.1	17.6	2.71	2.35
L74	189.5	26.9	4.15	2.19
L70	334.1	24.9	3.84	1.15
X81	100.0	16.6	2.57	2.57

## CONCLUSIONS

This study involves the determination of surface chemistry effects of oxide scale and coating composition on the hot corrosion life of selected alloys in the coated and uncoated conditions in burner rig tests. The burner rig data will be used to develop a hot corrosion life prediction model, to embrace both coating/alloy composition variables and environmental variables. Evaluation of service-run turbine components, which have suffered hot corrosion, establishes a baseline of microstructures and the extent of metal degradation as a function of operating history. This evaluation has been completed and was reported previously.

Burner rig test conditions are: 900° C, hourly thermal cycling, 0.5 ppm sodium as NaCl in the gas stream, velocity 0.3 Mach. The alloys are Udimet 700, René 80, uncoated and with RT21, Codep, or NiCoCrAlY coatings. These tests, up to 1000 hours, have been completed for specimens in the as-processed condition and after aging at 1100° C in oxidizing or inert environments for times up to 600 hours.

Coil inductance changes, used for periodic nondestructive inspection of specimens, are useful in following the course of corrosion for uncoated alloys for which degradation is rather uniform and generally extensive. However such changes are much smaller and less instructive for coated specimens for which degradation is localized and failure is defined when the relatively thin ( $\leq 100$  µm) coating is breached.

Typical sulfidation was observed in all cases, structurally similar to that observed for service-run turbine components. Aging at 1100° C caused a severe decrease in hot corrosion life of RT21 and Codep coatings and a significant but less dramatic decrease in the life of the NiCoCrAlY coating. The extent of these decreases was much greater for all three coatings on U700 substrates than on René 80 substrates. Coating life decrease is predominantly a result of composition changes caused by coating/substrate interdiffusion rather than by surface oxidation.

## REFERENCES

- 1. Fryxell, R.E., "Effects of Surface Chemistry on Hot Corrosion Life," First Annual Report, Contract NAS3-23926 (National Aeronautics and Space Administration), NASA CR-174683 (General Electric R84AEB422), June 1984.
- 2. Deadmore, D.L., "Application of Induction Coil Measurements to the Study of Superalloy Hot Corrosion and Oxidation," NASA TM-83560, January 1984.

